Notebook Series -- Notebooks by Edison
Notebook, N-15-09-23

This notebook was used by Edison in September 1915. The entries pertain primarily to methods of obtaining additional phenol from "waste liquor," aurin, and other byproducts of chemical production. Included are experiments with carbonated solutions, benzol, and naphta. Also included is one page of notes, entitled "Submarine," on the chemical absorption of hydrogen, which probably relates to the evolution of hydrogen gas by the Edison storage batteries used on submarines. The front cover is labeled "Phenol CO2 1915." The pages are unnumbered. Approximately 15 pages have been used.

CO_2 Experiment Sept 23 1915

Passed CO_2 from H_2SO_4 & Limestone through one qrt of Dally Quenching liquid Reg filtered, took 9 Cubic feet

Seperated phenol by funnel — There was considerable white floating stuff clinging to bottom of floating phenol. Treated the phenol with 98% H_2SO_4 diluted 1/2 acid & water by bulk

There was considerable CO_2 given off so it will be necessary in practice on a large scale to add it gradually

The net phenol was 310 cc giving 8.4 per 100 cc

This would give for 1962 gal 1340 lbs Crude, but not so much if we consider the bulk of the sulphite filtered out in addition there is probably 20% water in the Crude Phenol only net 1072 whereas we

should get 1550 pure —
Treated 800 cc of waste
Liquor with Benzol,
then treated Benzol
with 33% NaOH & neutralized
it & got 11 cc phenol
this brings total to
360 cc phenol & on basis
1962 gals given
1555 lbs phenol —
The question is now
How much water
was this phenol —

I tried solution again with Benzol but very small amount phenol came off less than 1 cc no doubt if agitated more than 5 min as we do it in seperating funnel we would get it all —

I also took the waste liquor from 1st benzol neutralized it with ½ + ½ acid + got less than a ¼ of a cc in 200 cc

This shows if well agitated in Benzol + then well agitated with NaOH, we get all the dissolved phenol in the liquor

There is some doubt if this 1st gal was fully Carbonated —
2nd one is being made

On distilling only got
38 cc free good phenol
and of 50 cc
24% of Water —

or 273 net,

or 1291 lbs per fusion —
259 lbs loss —

This loss is either in not carbonating
Enough. Or fusion was at too low
a temperature,

4.7 lbs to Gal Benzol.

In this distillation
there is a little of muck
& considerable black

This phenol crude after acids was quite dark red — The residue weighed 6 grams — was brittle & hard —

This neared make about 80 cc — 5 of water 6 of residuum or 69 cc phenol
11 cc Water + Residuum —

Phenol is white crystalline

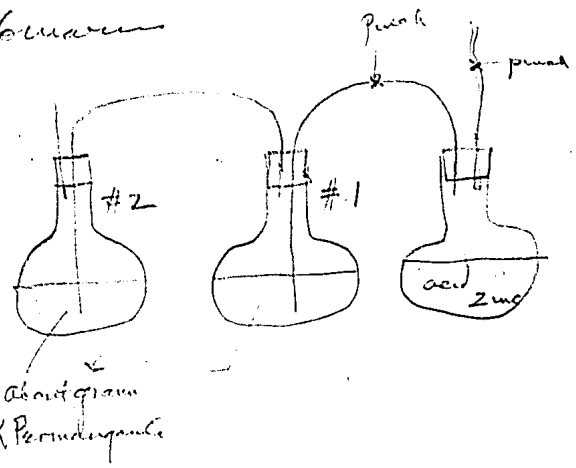
much Discoloring Phenol went to stronger acid treatment —

After treatment of phenol with 1/2 + 1/2 in Excess — The distillation showed some muck

I then ~~added~~ drew off phenol by seperating funnel & treated again with ~~acid~~ 1/2 + 1/2 H_2SO_4 — This threw out a larger quantity of precipitate that made the whole of the phenol very mucky — I again seperated the phenol by funnel & filtered it filters clear & leaves a white sediment on the funnel paper — only 5 cc water comes off 74 cc phenol
no much considerable aurin — purified — about 50%

I then treated some of the original Crude phenol with HCl This threw out Even more white stuff

Dubrunfaut



punch
punch
#2
#1
acid
zinc
about gram
K Permanganate

#1 is getting full of Manganese oxide
#2 only a little,

Its a pretty good absorber of Hydrogen

Try Peroxide Copper Lilly MnOH2 by Current & some endothermic substances —

Mean H_2SO_4 — It does not appear to want to settle out of the Phenol but may do so = Later 36 hours — While mixed some Phenol Clear & water also, where did it go ??

Think after treatment of phenol by strong acids that some acid left in — Think clean it in steel phenol wash with a Base that is so weak Phenol dont Combine with like Magnesia Oxide, Litharge =

Distilled some of the Avrin from Dallys 1st distillation of which he got 1200 lbs — At least 33% is Phenol & Can be got off by using one of Rdunnerth offs Little pills Dealing with NaOH filtering & Neutralizing

What is left is puffed stuff — may be valuable — Will have trouble get it out of pitch — Experiment

Cant get phenol from it by NaOH etc in the usual way must be a compound which is broken up in distilling giving up the phenol —

Daily amin

I seperated the phenol + put it in CC glass in Ice chest to see if it would crystalize without being distilled —

that was distilled off

36 hours in ice chest dedut Crystalize — found lot of salt at bottom of CC glass

Note CC necessary in Ice chest

The muck from big still distilled in iron pot, 1st trial got 123 cc liquid —

2nd with 1% Carbonate soda foamed terribly only got 100 cc liquid & it was mostly black JHSA suggestion is bad —

This muck was probably thicker than 1st muck

Have sent for fresh lot,

Masons Carbonating in glass tube, got 370 cc Crude phenol removed 50 cc — this left 320 cc but there was 10 cc mud at bottom so only 310 cc

Added 61 cc of 1/2+1/2 acid this brought out 109 cc

of water in addition to the 61cc
making 170cc clear liquor
acid water
The phenol was then drawn off
& filtered - it amounted
to _____ filter paper holds
11cc by weight.

Benzoled 300cc of the liquor
from which the Phenol was
removed from - this gave
2cc or 25cc per gallon
after 1/2 + 1/2 wash

306cc per gal - 89% by
distilling to pure Phenol

272 CP Phenol
per gallon by Carbonating

Took 50 cc of lots containing liquor after Bengaline a point in Bromine — great,

Tribromophenol contains 28% phenol —

The Tribromophenol weighed 1.22 grams — 28% gives 341 milligrams phenol —

There is 151400 50cc lots in one fusion — this gives 51627 grams phenol or 113.7 lbs lost —

After Benzolizing & neutralizing the Alkaline Liquor & Benzolized it again got no phenol – not a trace,

Spent Carbonated Liquor after Crude Benzol taken off Benzolized with 2 of Alkali & 1 Benzol & shook by hand 10 mins then Benzol treated with 2 KOH 2 Li gave no more than my other Experiment To wit scant 2 cc phenol

Using 300 cc Alkaline Liquor neutralized with 1/5 & 1/2 It got very hot,

Evidently Benzol only taken out a little more than 1/2 The phenol that is in an Alkaline Liquor Carbonated as previously shown by Bromine test,

Must try other things than Benzol –

1/5 ~~[illegible]~~ Benzol is apparently as good as ~~[illegible]~~ 1/3 of Benzol

Andrews test shows 4.5%
Sodium Chloride in our
Benzsulfonate of Soda

Solvent Naphta seems to
be as good as Benzol for
removing phenol from
Waste liquor

300 cc Tube Carbonated
liquor gives 2 to 2.25 cc
phenol which is a shade
better than Benzol

1/2 + 1/2 is liable to Brown
the solvent Naphta
also Soda takes out a

Something from Solvent
Naphta that 1/2 + 1/2 Brown

So free use Solvent
Naphta if shaved 6 m
could washed with
Soda —